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# SOME EFFECTS OF FILTRATION ON THE DETERMINATION OF NUTRIENTS IN FRESH AND SALT WATER[1]

ABSTRACT

The nutrient concentration of samples of water often changes significantly during filtration. Six types of filters and four nutrients in fresh- and also salt-water solution were tested. Two major sources of variation were elution of nutrients from the filters by the water samples and, conversely, adsorption of nutrients from the sample by the filter.

Samples of water containing suspended materials are often filtered, diluted with water, and then analyzed by standardized techniques. Errors may be introduced during the preanalysis treatment, especially during filtration. We show here possible effects of sample filtration on the reliability of four nutrient determinations in fresh and salt water.

The development of errors during filtration is not surprising when one considers the characteristics of filtering materials. Sandell (1950) stated that quantitative filter papers always contain metal contamination and also may adsorb such metals as lead and copper. Jenkins (1968) stated that Millipore filters (47 mm) were found to contain about 1.3 $\mu$g P/filter of which about 1 $\mu$g could be washed out. Robinson (1968) investigating the role of contamination in the trace element analysis of seawater found that materials used in the collection and treatment of water samples often contain high levels of impurities of various trace metals. In addition, Riley (1965) found that glass and plastic materials, because of their distorted and broken bond constructions, are able to adsorb ions from solution and that glass can act both as an anion exchanger and as a cation exchanger.

## PROCEDURE

Specific effects of filtration were determined by conducting analyses for $PO_4$, $NO_2$, $NO_3$, and $NH_3$ on filtered and unfiltered portions of freshwater and salt-water samples. The freshwater was distilled-deionized-distilled tapwater; the salt water, which had a salinity of about 20‰, was taken from an estuarine area (East Lagoon) on Galveston Island. Each salt-water sample (22.5 liters) was clarified by leaving it undisturbed for 3 days; a preservative (mercuric chloride) was added to minimize changes during sedimentation. Each sample was further clarified, by carefully siphoning off the upper 4 liters of water and discarding the remainder, to increase the probability that aliquots taken subsequently would be representative of the sample.

Each 4-liter sample was analyzed at either 5 or 6 concentration levels before and after filtration. Portions of the clarified samples were used as "zero-level" samples (i.e. no additions of chemicals were made). The remaining levels were attained by adding calculated amounts of sodium nitrate, sodium nitrite, ammonium sulfate, or potassium phosphate.

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Portions (100 ml) of samples of clarified lagoon water at each test level were filtered through a set of washed filters and analyzed; a second series was filtered through unwashed filters and analyzed. All samples, including an unfiltered series, were analyzed in triplicate.

The preparation of samples of fresh (distilled) water was identical except that the sedimentation and preservation steps were eliminated.

Six filter materials were used: 1) untreated filter paper (Whatman No. 1, 18.5-cm diameter); 2) acid-washed filter paper (Whatman No. 40, 18.5-cm diameter); 3) borosilicate glass wool with fibers ranging from 5–8 $\mu$; 4) Millipore filter (type AA, pore size, 0.8 $\mu$, 47-mm diameter); 5) cellulose acetate microporous membrane filter (Gelman GA 4, pore size, 0.8 $\mu$, 10.2-cm diameter); 6) sintered glass disk (8.9-cm diameter, pores ranging from 10–15 $\mu$). Except for the sintered glass disk, each filter was used only once. The paper and membrane filters were folded to fit 60° glass funnels; the membranes were softened in distilled water before folding. The glass wool filters were prepared by packing 2.5 g of the fibers into funnels tightly enough so that a portion of the wool extended into the stem of the funnel. The sintered glass disk was cleaned before each filtration by filtering through it, with the aid of suction, 100 ml of concentrated HCL and twenty 100-ml portions of distilled water.

The filters not washed before filtration were used as they were taken from their respective packing containers, except for the membrane filters which had to be softened before folding. Sintered glass disks were not included in the unwashed series.

The filters that were washed were rinsed with distilled water as follows: 200 ml of water were filtered through the glass wool and the paper filters. Wash water remaining in the glass wool was virtually eliminated by pressure applied with a porcelain pestle and then by a 50-ml rinse with a portion of the sample to be filtered. The membrane filter was washed by filtering 50 ml of distilled water through it. The sintered glass filter was assumed to be sufficiently washed after receiving the treatment mentioned, and the Millipore filter, by filtering 100 ml of distilled water through it with the aid of suction.

All analyses were performed by colorimetric procedures in an electrophotometer, nitrate by the method described by Marvin (1955), nitrite using the method of Bendschneider and Robinson (1952), ammonia by the method of Solórzano (1969), and inorganic phosphate by the procedure in Strickland and Parsons (1968).

The results of the chemical analyses and the means for each filter type are shown in Tables 1–4. The discussions of filter effects are based on these means.

Absorbance values were converted to $\mu$g-atom/liter units using calibration curves constructed from the unfiltered sample data that accompanied each series of filtered sample data. No attempt was made to correct for systematic errors resulting from reagent blanks, or for the natural occurrence in lagoon water of the nutrient for which the water was analyzed; these errors account for differences between actual values recorded for some of the test levels and their labeled values. They were systematic within groups so they did not interfere with the detection of filter effects.

The total effect of filtration on the chemical determinations was examined using a two-way analysis of variance for data with repeated measures. Classifications were levels of the chemical introduced into the solution and filter types. Both levels and filter types were considered to be fixed treatments. The differences between individual filter treatment means were estimated using Tukey's "W" test.

## RESULTS AND DISCUSSION

In the analysis for each set of data, the $F$ value for level was highly significant. This result was expected; in fact, any series of tests for which the difference

TABLE 1. *Analyses of filtered* and unfiltered samples for phosphate (in μg-atom $PO_4$-P/liter). Tukey's "W" values: salt-water washed filters, 0.107; salt-water unwashed filters, 0.172; freshwater washed filters, 0.073; freshwater unwashed filters, 0.080*

| Test level | Washed filters | | | | | | | Unwashed filters | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $F_0$ | $F_1$ | $F_2$ | $F_3$ | $F_4$ | $F_5$ | $F_6$ | $F_0$ | $F_1$ | $F_2$ | $F_3$ | $F_4$ | $F_5$ |
| | | | | | | Salt water | | | | | | | |
| 0 | 5.43 | 5.10 | 5.00 | 5.00 | 5.03 | 5.20 | 4.67 | 5.70 | 5.57 | 5.47 | 5.67 | 5.60 | 5.60 |
| 1 | 6.37 | 5.97 | 5.93 | 6.00 | 5.97 | 6.07 | 5.60 | 6.37 | 5.97 | 6.13 | 6.23 | 6.23 | 6.27 |
| 2 | 7.37 | 6.97 | 6.97 | 7.07 | 6.87 | 7.13 | 6.70 | 7.60 | 7.33 | 7.37 | 7.60 | 7.43 | 7.30 |
| 3 | 8.43 | 8.10 | 7.87 | 8.07 | 7.90 | 8.00 | 7.57 | 8.67 | 8.70 | 8.70 | 8.90 | 8.70 | 8.73 |
| 4 | 9.43 | 8.90 | 8.83 | 9.10 | 8.90 | 9.03 | 8.57 | 9.53 | 9.40 | 9.40 | 9.53 | 9.10 | 9.57 |
| 5 | 9.90 | 9.90 | 9.73 | 9.97 | 9.80 | 9.90 | 9.40 | 10.63 | 10.43 | 10.33 | 10.70 | 10.23 | 10.47 |
| Mean | 7.822 | 7.489 | 7.389 | 7.533 | 7.411 | 7.555 | 7.083 | 8.083 | 7.900 | 7.900 | 8.106 | 7.883 | 7.989 |
| | | | | | | Freshwater | | | | | | | |
| 0 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 | 0.00 | 0.00 | 0.00 | 0.07 | 0.20 | 0.27 | 0.10 | 0.20 |
| 1 | 0.96 | 1.00 | 0.90 | 1.00 | 1.00 | 0.97 | 1.03 | 1.00 | 1.07 | 1.20 | 1.20 | 1.03 | 1.40 |
| 2 | 1.97 | 2.07 | 1.90 | 2.07 | 2.00 | 1.97 | 2.10 | 2.00 | 2.07 | 2.10 | 2.13 | 2.00 | 2.40 |
| 3 | 2.96 | 2.90 | 2.93 | 3.00 | 2.97 | 2.97 | 3.07 | 3.00 | 3.03 | 3.13 | 3.13 | 2.97 | 3.33 |
| 4 | 4.00 | 3.93 | 3.94 | 4.03 | 3.90 | 3.97 | 4.03 | 4.07 | 4.11 | 4.13 | 4.13 | 3.97 | 4.20 |
| 5 | 4.97 | 4.83 | 4.84 | 4.83 | 5.03 | 4.93 | 4.83 | 5.00 | 5.00 | 5.10 | 5.10 | 4.83 | 4.90 |
| Mean | 2.478 | 2.465 | 2.417 | 2.489 | 2.489 | 2.467 | 2.511 | 2.511 | 2.550 | 2.644 | 2.661 | 2.483 | 2.739 |

* Filter type: $F_0$ = none; $F_1$ = untreated paper (Whatman No. 1); $F_2$ = acid-washed paper (Whatman No. 40); $F_3$ = borosilicate glass wool; $F_4$ = Millipore; $F_5$ = membrane (Gelman GA-4); $F_6$ = sintered glass.

TABLE 2. *Analyses of filtered* and unfiltered samples for nitrite (in μg-atom $NO_2$-N/liter). Tukey's "W" values: salt-water washed filters, 0.050; salt-water unwashed filters, 0.017; freshwater washed filters, 0.027; freshwater unwashed filters, 0.029*

| Test level | Washed filters | | | | | | | Unwashed filters | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $F_0$ | $F_1$ | $F_2$ | $F_3$ | $F_4$ | $F_5$ | $F_6$ | $F_0$ | $F_1$ | $F_2$ | $F_3$ | $F_4$ | $F_5$ |
| | | | | | | Salt water | | | | | | | |
| 0.0 | 0.580 | 0.450 | 0.410 | 0.433 | 0.340 | 0.477 | 0.313 | 0.427 | 0.510 | 0.280 | 0.717 | 0.170 | 0.293 |
| 0.3 | 0.877 | 0.680 | 0.700 | 0.667 | 0.633 | 0.890 | 0.640 | 0.727 | 0.786 | 0.557 | 1.137 | 0.720 | 0.573 |
| 0.6 | 1.177 | 1.077 | 1.040 | 0.970 | 0.877 | 1.297 | 0.933 | 1.033 | 1.183 | 0.910 | 1.370 | 0.820 | 0.990 |
| 0.9 | 1.470 | 1.313 | 1.293 | 1.190 | 1.213 | 1.553 | 1.150 | 1.340 | 1.590 | 1.277 | 1.720 | 1.210 | 1.383 |
| 1.2 | 1.770 | 1.560 | 1.567 | 1.556 | 1.483 | 1.580 | 1.477 | 1.633 | 1.860 | 1.550 | 2.270 | 1.467 | 1.650 |
| 1.5 | 2.077 | 1.913 | 1.867 | 1.933 | 1.810 | 2.017 | 1.790 | 1.930 | 2.180 | 1.850 | 2.430 | 1.773 | 1.940 |
| Mean | 1.325 | 1.166 | 1.146 | 1.124 | 1.059 | 1.302 | 1.051 | 1.182 | 1.352 | 1.071 | 1.607 | 1.027 | 1.138 |
| | | | | | | Freshwater | | | | | | | |
| 0.0 | 0.003 | 0.090 | 0.030 | 0.120 | 0.070 | 0.087 | 0.053 | 0.053 | 0.327 | 0.123 | 0.703 | 0.127 | 0.353 |
| 0.3 | 0.310 | 0.367 | 0.310 | 0.340 | 0.347 | 0.357 | 0.333 | 0.347 | 0.600 | 0.393 | 0.923 | 0.367 | 0.607 |
| 0.6 | 0.610 | 0.657 | 0.600 | 0.620 | 0.637 | 0.650 | 0.620 | 0.650 | 0.890 | 0.700 | 1.943 | 0.720 | 0.847 |
| 0.9 | 0.907 | 0.920 | 0.907 | 0.927 | 0.913 | 0.907 | 0.900 | 0.943 | 1.243 | 0.977 | 1.967 | 0.960 | 1.147 |
| 1.2 | 1.217 | 1.187 | 1.167 | 1.197 | 1.193 | 1.193 | 1.223 | 1.253 | 1.480 | 1.283 | 2.540 | 1.283 | 1.440 |
| 1.5 | 1.503 | 1.510 | 1.467 | 1.507 | 1.497 | 1.473 | 1.487 | 1.553 | 1.793 | 1.580 | 2.733 | 1.613 | 1.743 |
| Mean | 0.758 | 0.788 | 0.747 | 0.785 | 0.776 | 0.778 | 0.769 | 0.800 | 1.056 | 0.842 | 1.802 | 0.845 | 1.023 |

* Filter type: same as Table 1.

TABLE 3. *Analyses of filtered* and unfiltered samples for nitrate (in μg-atom $NO_3$-N/liter). Tukey's "W" values: salt-water washed filters, 0.415; salt-water unwashed filters, 0.686; freshwater washed filters, 0.440; freshwater unwashed filters, 0.316*

| Test level | Washed filters | | | | | | | Unwashed filters | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $F_0$ | $F_1$ | $F_2$ | $F_3$ | $F_4$ | $F_5$ | $F_6$ | $F_0$ | $F_1$ | $F_2$ | $F_3$ | $F_4$ | $F_5$ |
| | | | | | | Salt water | | | | | | | |
| 0 | 0.00 | 2.73 | 0.53 | 1.57 | 1.10 | 3.97 | 0.10 | 0.10 | OFF SPACE | 3.17 | 9.83 | 0.57 | 11.37 |
| 2 | 2.33 | 6.80 | 1.93 | 2.57 | 3.33 | 4.20 | 2.00 | 2.03 | | 1.87 | 13.90 | 3.87 | 9.23 |
| 4 | 3.73 | 7.03 | 3.83 | 4.00 | 5.43 | 6.27 | 4.03 | 3.97 | | 3.73 | 12.00 | 5.93 | 11.40 |
| 6 | 5.90 | 9.93 | 5.17 | 5.30 | 7.07 | 5.70 | 5.67 | 5.93 | | 6.60 | 9.20 | 7.73 | 10.70 |
| 8 | 8.00 | 13.93 | 5.07 | 5.33 | 8.60 | 6.60 | 6.60 | 7.67 | | 8.10 | 10.57 | 9.03 | 9.70 |
| Mean | 3.993 | 8.087 | 3.307 | 3.753 | 5.107 | 5.347 | 3.680 | 3.940 | | 4.693 | 11.100 | 5.427 | 10.480 |
| | | | | | | Freshwater | | | | | | | |
| 0 | 0.13 | 1.06 | 0.23 | 0.30 | 1.07 | 0.87 | 0.43 | 0.07 | OFF SPACE | 0.50 | 8.53 | 3.20 | 10.37 |
| 2 | 2.13 | 9.63 | 1.87 | 2.03 | 3.43 | 3.03 | 1.80 | 2.03 | | 2.50 | 4.97 | 3.80 | 11.33 |
| 4 | 4.20 | 10.07 | 4.27 | 4.67 | 6.13 | 5.33 | 5.10 | 3.93 | | 4.37 | 6.90 | 5.33 | 11.83 |
| 6 | 5.97 | 9.57 | 6.13 | 6.27 | 7.30 | 9.77 | 6.10 | 6.07 | | 6.80 | 8.77 | 7.47 | 12.67 |
| 8 | 8.20 | 10.67 | 6.60 | 7.70 | 8.63 | 9.77 | 9.73 | 8.10 | | 8.87 | 10.60 | 8.80 | 13.27 |
| Mean | 4.127 | 8.200 | 3.820 | 4.193 | 5.313 | 5.753 | 4.633 | 4.040 | | 4.607 | 7.953 | 5.720 | 11.893 |

* Filter type: same as Table 1.

TABLE 4. *Analyses of filtered* and unfiltered samples for ammonia (in μg-atom $NH_4$-N/liter). Tukey's "W" values: salt-water washed filters, 0.833; salt-water unwashed filters, 0.248; freshwater washed filters, 0.420; freshwater unwashed filters, 0.220*

| Test level | Washed filters | | | | | | | Unwashed filters | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $F_0$ | $F_1$ | $F_2$ | $F_3$ | $F_4$ | $F_5$ | $F_6$ | $F_0$ | $F_1$ | | $F_2$ | $F_3$ | $F_4$ | $F_5$ |
| | | | | | | Salt water | | | | | | | | |
| 0 | 1.60 | 1.70 | 9.23 | 1.63 | 2.20 | 2.90 | 2.27 | 15.87 | 18.60 | | | 17.37 | 20.00 | |
| 2 | 3.63 | 3.77 | 9.83 | 4.30 | 3.76 | 4.17 | 3.67 | 18.40 | 19.17 | | | 18.70 | 24.20 | |
| 4 | 5.57 | 5.67 | 11.17 | 5.70 | 5.50 | 5.43 | 5.90 | 20.13 | 21.30 | | | 24.10 | 25.10 | |
| 6 | 7.67 | 7.53 | 10.10 | 7.23 | 7.53 | 8.27 | 7.30 | 22.10 | 23.50 | | | 22.73 | 27.30 | |
| 8 | 9.60 | 9.27 | 17.50 | 9.20 | 9.33 | 9.60 | 9.03 | 24.10 | 25.47 | | | 25.60 | 28.20 | |
| 10 | 11.47 | 12.03 | 16.97 | 11.37 | 11.60 | 11.43 | 11.13 | 26.00 | 27.17 | | | 28.80 | 29.83 | |
| Mean | 6.589 | 6.661 | 12.467 | 6.572 | 6.639 | 6.967 | 6.550 | 21.10 | 22.533 | | | 22.883 | 25.772 | |
| | | | | | | Freshwater | | | | | | | | |
| 0 | 0.00 | 1.80 | 6.90 | 1.33 | 2.53 | 2.00 | 0.80 | 0.00 | 1.30 | | 13.23 | 0.87 | 3.73 | |
| 2 | 2.03 | 2.03 | 8.33 | 1.20 | 4.70 | 4.80 | 1.47 | 2.03 | 1.97 | | 17.63 | 3.07 | 6.57 | |
| 4 | 4.00 | 4.43 | 7.73 | 4.20 | 6.47 | 5.80 | 3.03 | 3.97 | 2.67 | | 16.20 | 2.30 | 8.60 | |
| 6 | 6.00 | 5.67 | 10.88 | 5.43 | 7.93 | 8.00 | 3.80 | 6.03 | 4.23 | | 18.80 | 2.60 | 9.33 | |
| 8 | 7.97 | 7.73 | 10.07 | 7.00 | 9.10 | 9.30 | 4.33 | 8.00 | 5.70 | | 22.27 | 5.00 | 9.60 | |
| 10 | 9.97 | 8.60 | 11.80 | 9.13 | 10.33 | 10.77 | 4.47 | 10.03 | 7.60 | | 22.73 | 12.47 | 13.13 | |
| Mean | 4.994 | 5.044 | 9.283 | 4.716 | 6.844 | 6.778 | 2.983 | 5.011 | 3.911 | | 18.478 | 4.383 | 8.494 | |

OFF SPACE

OFF SPACE

* Filter type: same as Table 1.

NOTES

between levels was not detectable would be subject to question.

The level × filter type interaction was highly significant in each analysis performed. This indicates that the effects of the filter often change with the level of nutrient present within the range under consideration. This was true of both washed and unwashed filters.

Statistically significant differences between filter types were observed for each nutrient; that is, in each experiment at least one filter had an adverse effect on the results of the determinations. The results are presented as two groups of means per determination—the washed group and the unwashed group. The "W" values included in Tables 1–4 indicate the least significant difference at the 5% level within each set of means. To simplify comparisons, means that showed no evidence of filter effect are underlined.

Where filtered means were significantly greater than the unfiltered mean within a series of data, nutrients had apparently been eluted from contaminated filters. Similarly, where means of filtered samples were significantly less than the unfiltered mean in a series of data, nutrients had apparently been adsorbed by the filter. Sample elution was more noticeable among data from unwashed groups and particularly unwashed freshwater groups. With the exception of one freshwater $NH_3$ mean ($F_6$: Table 4), filter adsorption was limited to the salt-water samples and particularly the washed groups. Apparently effects of sample elution that tend to neutralize adsorption effects were more prevalent among unwashed means.

Some suspended matter remained in the salt-water samples after clarification, evident from traces of residue that remained on some of the filters after use. We believe this material was responsible for much of the filter adsorption of nutrients from the salt-water samples by adsorbing some of the added (as well as naturally occurring) nutrients within the samples. Nutrients thus adsorbed were later removed from the sample as part of the suspended matter during subsequent filtrations.

TABLE 5. *Summation of results showing filter*-nutrient-water type combinations that do not alter the reliability of the nutrient determination*

| Nutrient | Washed filters | Unwashed filters |
|---|---|---|
| | Freshwater samples | |
| Nitrite | $F_2$, $F_3$, $F_4$, $F_5$, $F_6$ | |
| Nitrate | $F_2$, $F_3$ | |
| Ammonia | $F_1$, $F_3$ | |
| Phosphate | $F_1$, $F_2$, $F_3$, $F_4$, $F_5$, $F_6$ | $F_1$, $F_4$ |
| | Salt-water samples | |
| Nitrite | $F_5$ | |
| Nitrate | $F_3$, $F_6$ | |
| Ammonia | $F_1$, $F_3$, $F_4$, $F_5$, $F_6$ | |
| Phosphate | | $F_3$, $F_5$ |

* Filter type: same as Table 1.

Ion exchange may have been responsible for some of the differences. An example of this exchange was indicated, we believe, by the removal of significant amounts of $NH_3$ from the (particulate-free) freshwater $NH_3$ samples by the unwashed $F_1$ and $F_3$ filters, and the washed $F_6$ filter.

For the sake of clarity, filter-nutrient-water type combinations unaffected by filtration are given in Table 5. Successful filtration always involved washed filters except in the case of $PO_4$. In this determination, $F_1$ and $F_4$ were not contaminated with $PO_4$ and thus they performed equally well, washed or unwashed, with freshwater samples. Because these filters did not contain available $PO_4$, their ability to adsorb it from salt water was not neutralized by elution, and thus they exhibited a maximum adsorption effect (Table 1). In contrast, unwashed filters $F_3$ and $F_5$ contained enough $PO_4$ (contaminant) to counteract effectively their ability to adsorb $PO_4$ from salt-water samples.

Because this investigation was based on samples and conditions rarely, if ever, encountered under field conditions, the nutrient determinations were sufficiently reliable (indicated by Tukey's "W" values)

to enable us to detect subtle filter effects. Under field conditions the reliability of determinations would vary from those of this investigation. As the reliability of a determination increases, the effects of filtration become more critical. If, for example, the reliability of the $PO_4$ determination were reduced enough to increase Tukey's "W" from 0.172 to 0.200 (Table 1), none of the effects of unwashed filters on the $PO_4$ concentration of the salt-water samples would have been judged significant.

Our data show that, based on the precision of our analyses, each nutrient tested was unaffected by at least one filter and that $PO_4$ was the only nutrient unaffected by unwashed filters ($F_3$ and $F_5$).

The salt-water data contain the applicable conclusions of our study. The freshwater data merely demonstrate the extent of filter contamination and the benefits of filter washing.

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